

UCO 600 PCT/MO 10/01863 17 DEC 2001

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| Express Mail No.: EV 042503410 US | Date: December 17, 2001 |
| I hereby certify that this correspondence is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Commissioner of Patents and Trademarks, Washington, D.C. 20231. | |
| <u>Antonella Fusillo</u> | <u><i>Antonella Fusillo</i></u> |
| (Name of person mailing paper or fee) | (Signature) |

| | | |
|--|---|---|
| TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371 | | Attorney's Docket No: SCHÖNEFELD |
| INTERNATIONAL APPLICATION NO. PCT/DE00/01885 | INTERNATIONAL FILING DATE 14 June 2000 | PRIORITY DATE CLAIMED 18 June 1999 |
| TITLE OF INVENTION VERFAHREN ZUR DYNAMISCH-CHEMISCHEN HERSTELLUNG VON DIAMENTARTIGEN KOHLENSTOFFSTRUKTUREN, DIAMANTARTIGE KOHLENSTOFFSTRUKTUREN UND VERWENDUNGEN VON DIAMANTARTIGEN KOHLENSTOFFSTRUKTUREN | | |
| APPLICANT(S) FOR DO/EO/US CHRISTA SCHÖNEFELD AND TEJAS SCHÖNEFELD | | |

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☐ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ Original or facsimile of an oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A FIRST preliminary amendment.
☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information: Form PCT/IB/308

| | | | |
|---|--------------|-------------------------------|-----------------------|
| U.S. APPLICATION NO. (if known, see 37 CFR 1.5) | | INTERNATIONAL APPLICATION NO. | ATTORNEY'S DOCKET NO. |
| 10/018634 | | PCT/DE00/01885 | SCHÖNEFELD |
| 17. <input checked="" type="checkbox"/> The following fees are submitted : BASIC NATIONAL FEE (37 C.F.R. 1.492(a)(1)-(5): | | | |
| <input checked="" type="checkbox"/> For filing with EPO or JPO search report (37 C.F.R. 1.492(a)(5)) | | | \$ 890.00 |
| <input type="checkbox"/> International preliminary examination fee paid to USPTO (37 C.F.R. 1.492(a)(1)) | | | \$ 710.00 |
| <input type="checkbox"/> No international preliminary examination fee paid to USPTO (37 C.F.R. 1.492(a)(2)) but international search fee paid to USPTO (37 C.F.R. 1.445(a)(2)) | | | \$ 740.00 |
| <input type="checkbox"/> Neither international preliminary examination fee paid to USPTO (37 C.F.R. 1.492(a)(3)) nor international search fee paid to USPTO (37 C.F.R. 1.445(a)(2)) | | | \$1,040.00 |
| <input type="checkbox"/> International preliminary examination fee paid to USPTO (37 C.F.R. 1.492(a)(4)) and all claims satisfied provisions of PCT Articles 33(2)-33(4) | | | \$ 100.00 |
| Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)). | | | |
| Claims | Number Field | Rate | |
| Total Claims | -20 | x \$ 18.00 | |
| Independent Claims | -3 | x \$ 84.00 | |
| Multiple dependent claims (if applicable) | | x \$280.00 | |
| TOTAL OF ABOVE CALCULATIONS | | | \$890.00 |
| <input checked="" type="checkbox"/> Applicant claims small entity status pursuant to 37 C.F.R. 1.27. Reduction by 1/2 for filing by small entity. | | | |
| SUBTOTAL | | | \$445.00 |
| Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date 37 CFR 1.492(f). | | | |
| TOTAL NATIONAL FEE | | | \$445.00 |
| Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property + | | | \$ 0.00 |
| TOTAL FEES ENCLOSED | | | \$445.00 |
| Amount to be refunded charged | | | |

a. ☒ A check in the amount of **\$445.00** to cover the above fees is enclosed.

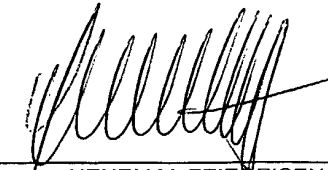
b. ☐ Please charge my Deposit Account No. **06-0502** in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. **06-0502**. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

Send all correspondence to:

HENRY M. FEIEREISEN
 350 Fifth Avenue
 Suite 3220
 New York, N.Y. 10118
 (212) 244-5500
 Date: December 17, 2001


 HENRY M. FEIEREISEN
 Registration No. 31,084

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE


Docket No.: SCHÖNEFELD

| | |
|-----------------------------------|---|
| In re Application of: |) |
| CHRISTA SCHÖNEFELD ET AL. |) |
| U.S. Appl. No.: 10/018,634 |) |
| Filed: December 17, 2001 |) |
| For: METHOD FOR DYNAMIC-CHEMICAL |) |
| PRODUCTION OF ADAMANTINE CARBON |) |
| STRUCTURES, ADAMANTINE CARBON |) |
| STRUCTUTES AND USES OF ADAMANTINE |) |
| CARBON STRUCTURES |) |

PRELIMINARY AMENDMENT

Commissioner for Patents
Washington, D.C. 20231

S I R:

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| I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to the Assistant Commissioner for Patents, Washington, D C 20231, on <u>May 28, 2002.</u> | |
| (Date) | |
| <u>Ursula B. Day</u> | |
| (Name of Registered Representative) | |
|  Signature | <u>May 28, 2002</u> Date of Signature |

Preliminary to the first Official Action in the above-entitled application, please amend the application as follows.

The Commissioner is hereby also authorized to charge any fees which may be required during the pendency of this application, including any patent application processing fees under 37 C.F.R. 1.17, and any filing fees under 37 C.F.R. 1.16, including presentation of extra claims, or credit any overpayment to Deposit Account No: 06-0502.

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CLEAN VERSION OF AMENDED CLAIMS

4. The method as claimed in Claim 2, characterised in that the carbon donor system has organic carbon compounds.
9. A use of adamantane carbon structures, in particular as claimed in claim 1 for surface treatment of hard materials.
10. The use of adamantane carbon structures, in particular as claimed in claim 1, as electrical insulator.
11. The use of adamantane carbon structures, in particular as claimed in claim 1, as thermal transfer medium.
12. The use of adamantane carbon structures, in particular as claimed in claim 1, as addition to a suspension, dispersion, emulsion, a spray, a paste, a fat or a wax.

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VERSION WITH MARKINGS TO SHOW CHANGES MADE:

IN THE SPECIFICATION:

Before paragraph [0001], add the heading --BACKGROUND OF THE INVENTION--

Before paragraph [0009], add the heading --SUMMARY OF THE INVENTION--.

Before paragraph [0022], add the heading --BRIEF DESCRIPTION OF THE
DRAWING--.

Before paragraph [0031], add the heading --DETAILED DESCRIPTION OF
PREFERRED EMBODIMENTS--.

Page 27, after the heading "CLAIMS" and before the first claim add --What is claimed
is:--.

IN THE CLAIMS:

Amend the following claims:

4. The method as claimed in Claim 2 ~~[or 3]~~, characterised in that the carbon donor system has organic carbon compounds.
9. A use of adamantane carbon structures, in particular as claimed in claim 1 ~~[any one of the foregoing claims]~~ for surface treatment of hard materials.

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10. The use of adamantine carbon structures, in particular as claimed in claim 1 [~~any one of Claims 1 to 8~~], as electrical insulator.
11. The use of adamantine carbon structures, in particular as claimed in claim 1 [~~any one of Claims 1 to 8~~], as thermal transfer medium.
12. The use of adamantine carbon structures, in particular as claimed in claim 1 [~~any one of Claims 1 to 8~~], as addition to a suspension, dispersion, emulsion, a spray, a paste, a fat or a wax.

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REMARKS

This Amendment is submitted preliminary to the issuance of an Office Action in the present application.

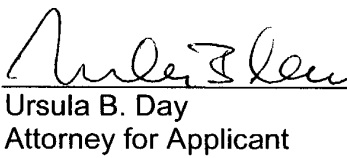
Applicant has amended the claims to eliminate the multiple dependencies thereof.

In addition, applicant has amended the specification to provide headings.

When the Examiner takes this application up for action, s/he is requested to take the foregoing into account.

The Commissioner is hereby authorized to charge fees which may be required, or credit any overpayment to Deposit Account No. 06-0502.

Respectfully submitted,

By: 
Ursula B. Day
Attorney for Applicant
Reg. No. 47,296

Date: May 28, 2002
350 Fifth Avenue
Suite 3220
New York, N.Y. 10118
(212) 244-5500
UBD:be

METHOD FOR DYNAMIC-CHEMICAL PRODUCTION OF
ADAMANTINE CARBON STRUCTURES, ADAMANTINE
CARBON STRUCTURES AND USES OF ADAMANTINE
CARBON STRUCTURES



[0001] The present invention relates to methods for dynamic-chemical production of adamantine carbon structures, in which a hybrid carbon phase is placed into a closed container and brought to a chemical reaction with an energy carrier to form disperse condensed carbon as a reaction product. The invention also relates to various adamantine carbon structures and uses of such carbon structures.

[0002] Disperse condensed carbon is produced as a phase-changed carbon structure, in particular in terms of synthetic diamond manufacture. Apart from static catalytic high-temperature pressure, impact pressure, physical (PVD) and chemical (CVD) and combined methods, so-called dynamic methods are also being employed which are based on chemical conversion of energy-rich materials and compounds and result in the development of hexagonal and cubic carbon structures predominantly in the form of disperse and ultradisperse systems.

[0003] The basis of dynamic chemical production of adamantine carbon structures is formed by the regularities which occur briefly with chemical

conversion of high-energy substances, predominantly with negative oxygen balance. They essentially comprise the fact that the condensed carbon released during chemical reaction of these substances and characterised by the generator gas reaction is subjected to such conditions that a phase change in higher structured crystal lattice structures can take place.

[0004] The chemical reaction of energy carriers containing carbon is generally achieved through explosive substances with negative oxygen balance and is carried out in closed high-pressure containers under conditions of an inert gas atmosphere. At the same time the atmospheric oxygen is to be eliminated as far as possible by vacuuming of the container system to obtain an inert gas atmosphere by means of special inert gases or their mixtures which are respectively under functional pressure. This inert gas atmosphere should counteract regraphiting of the higher structured carbon phases.

[0005] In technical and technological terms, however, these methods prove to be extremely expensive and uneconomical. Despite ultradisperse condensed carbon phases being provided to the reaction process only economically extremely low formation rates of approximately 8.0 % to 10.0 % mass fraction of the respectively produced and desired carbon structures are achieved, relative to the mass of the reaction or donor material being used. Technologies of this type are therefore industrially and economically irrelevant.

[0006] The higher structured carbon systems produced with this method contain up to 40 % hexagonal diamond structures (low-grade lonselite) and up to 30 % radiologically amorphous phases and residually cubic diamond components which nevertheless display a phase purity of not more than 85 % to 95 %. There are therefore mechanical quantities of various carbon structures with practically indefinable system properties, whose technical-industrial usability is to a large extent limited.

[0007] In addition, on the material surface these substance configurations contain a plurality of functional groups and carbon atoms with free bonds, which result in hard-to-define surface polarities, and thus impede or even make required cross-linking processes impossible in conjunction with other substances and materials.

[0008] The object of the present invention is to develop a method for dynamic chemical production of adamantane carbon structures, which enables economical production methods with simultaneously high phase purity. In addition, adamantane carbon structures with defined properties are to be provided and new uses for such materials are to be proposed.

[0009] According to the method the task is solved using a generic method, wherein the reaction product is subjected to an atomically hydrogen-supported low-

temperature plasma and a phase change of carbon combinations to high-purity cubic lattice structures is made.

[0010] Surprisingly, it has been shown that the use of an atomically hydrogen-supported low-temperature plasma leads to a very high process yield of high-purity cubic lattice structures.

[0011] Preferably, a carbon donor system is also introduced. This carbon donor system preferably has liquid or gaseous carbon compounds, whereby particularly good processing results were achieved especially with organic carbon compounds.

[0012] The finding of the invention is that hybrid carbon systems, primarily liquid carbon compounds, can be introduced in conjunction with condensed carbon resulting from the chemical conversion of energy carriers to a high-energy and brief chemical and thermogas dynamic process, such that on the one hand the formation of condensed carbon is supported stoichiometrically, and on the other hand the coalescence of already formed or deposited clusters from the chemical reaction of a used energy carrier is initiated and can be realised optimally in the complex system.

[0013] At the same time the chemical physical formation process is supported under conditions of atomically hydrogen-supported low-temperature

plasma in the presence of ^1H symbol 179 \f "Symbol" \s 12 $\square 9.12 \times 10^{17}$ to the extent that electron promotion with replenished hybridising occurs predominantly not according to a diffusion mechanism but according to a martensite mechanism.

[0014] This allows adamantine carbon structures to be produced economically on an industrial scale in nanoscale and microscale crystallite areas, as well as in cluster and polycrystalline structures with new production properties. The adamantine carbon structures thus produced can be technically formed, such that they create starting materials for presenting higher structured carbon systems, such as fullerene, hyper-fullerene, nano-tubes, onion-like carbons (OLC) and others.

[0015] A further object of the invention is adamantane carbon structures which are characterised in that the purity of the cubic diamond phase is 99 %, preferably 100 % (NJC Scan 1/X-Ray pattern). The crystallite sizes of the carbon structures are in the range between 5 nm and 50 nm (X-ray diffraction). Cluster orders of magnitude of 50 nm to 20 µm (scanning electron microscope) were achieved, as well as particle diameters in dispersion of 40 nm to 500 nm (photon correlation spectroscopy). Röntgen-amorphous phases do not occur.

[0016] Various parameters of the material can be varied or doped(cloning)using this method: specific surface values, specific magnetisation, zeta potential, specific electrical resistance, free energy with respect to vapour

[0019] They may not, however, fall below the range of 0.005 μ Ra and pole tip recessions (PTR) of 0.01 μ . Additionally, the industrial use of Mypolex configurations and other diamond high-performance systems is extremely cost-intensive and in the case of a series of technical applications is not optimal and does not lead to the desired aim.

[0020] However, the abovedescribed adamantine carbon structures according to the present invention can be produced economically and efficiently by dynamic chemical hybrid technologies and display a large number of specific properties which they produce compared to conventional industrial diamond configurations.

[0021] Due to the specific properties, in particular morphology and grain shape, as well as the specific surface characteristics and average zeta potential of the adamantine carbon structures according to the present invention finishing values of Ra = 2 nm to 10 nm and pole tip recessions of PTR = 0.5 nm to 2 nm can be achieved on superhard material surfaces. Also, polishing speeds of approximately 0.3 μ m to 5.0 μ m per minute are achievable, resulting in optimum, gentle smoothing effects.

[0022] Figures 1 to 4 illustrate the results of a nano sheen according to the present invention of a rough-ground surface made of silicon nitrite ceramic, as compared to high-performance diamond granulation. Figures 1 and 2 illustrate a

silicon nitride ceramic which was ground and then polished with a commercially available diamond suspension. Figures 3 and 4 illustrate by way of contrast a nano-high-end sheen with an aqueous-cationic, short-chain suspension made of adamantane carbon structures on a ground silicon nitride ceramic.

[0023] The lenticular (ogival) grain arrangement of the single-cell crystalline and the synthesis process, which guarantees a crystal-forming process from "below", are decisive inter alia for the beneficial use of adamantane carbon structures for surface processing of hard material surfaces. This means that the grain or the corresponding cluster configurations grow to a defined size and are not, as was common at one time, broken by means of milling procedures from larger grain structures, which inevitably leads to sharp contours and to the formation of coherent cleavage planes with simultaneously reduced compressive resistance.

[0024] The adamantane carbon structures according to the present invention are particularly suited to the following treatment methods:

[0025] treatment of high-strength ceramics, precious stones and special materials for elements and componentry in micro-electronics and high-performance electronics. The optimum polishing speed is between 0.3 μm and 5 μm per minute. precision polishes on metal-coated parts of a memory disc or metal mirrors,

treatment of parts and components made of polycarbonates such as spectacle lenses and similar,

treatment of optical, optronic and laser components and magnetic head systems,

treatment of orthopaedic and dental prostheses,

for micro-honing tools, miniature and precision ball bearings,

for mechanical sealing and sliding systems (such as inter alia pumps, valves, cylinders, pistons, bearings, bushes and surfaces of forming stamps),

metallurgical and crystallographic preparations,

treatment of elements and components made of polyacryl (for example, windows for aircraft cabins etc.) and contact lenses,

polished finishes of intricate and non-planar surfaces by means of grinding (rotofinishing) and planarising of elements used in heavy-duty and micro-electronics.

[0026] To achieve the desired distinguishing and efficiency characteristics the adamantine carbon structures according to the present invention are to be placed in specially designed media. Aqueous and organic water-soluble suspensions, emulsions, trays, fats, pastes and waxes are suitable as media.

[0027] A further advantageous use of adamantine carbon, in particular of the abovementioned carbon structures, is as electrical insulator. The adamantine carbon structures can also be utilised as thermal transfer media. It is particularly

beneficial if, apart from their high capacity for electrical insulation, the properties can be used for optimum transfer of thermal energy from one body to the other. This is a typical type of problem with lossy electronic components. The invention accordingly describes an advantageous use of adamantane carbon structures, predominantly in nano- and micrograin areas as electrical insulation or thermal transfer media. This is achieved by placing the carbon material into pastes, glues, paints, fats, solders and into composite material compounds primarily for technical-industrial use in areas of heavy-duty electronics, micro-electronics as well as electrotechnology and energetics.

[0028] With electronic componentry, semiconductor elements in particular, there is considerable reliance on temperature with respect to linearity and functionality. If it is assumed that for example barrier layer crystals made of germanium can be loaded in temperature ranges between 85 °C and 100 °C and the linearity of the structural elements is adequate often only in a limited range in terms of circuit technology, the generated heat must be diverted to the exterior to ensure readiness for service and to keep the semiconductor in a set temperature window.

[0029] The active exothermal surface is being enlarged these days by means of heat sinks. But since these high-performance components are not flat and therefore exhibit a certain roughness, they are connected by means of a so-called heat-conducting medium to the heat sink. At the same time these media

must have an extraordinarily high electrical insulating capacity and guarantee optimum adaptability to the corresponding surface without on the other hand showing flow behaviour.

[0030] Hitherto known pastes, fats, paints, glues, solders, film systems etc. whose thermal transfer properties are described by their specific and material-dependent conductivity, may absolutely not physically exceed electric conductances of 2.5 W/mK. Furthermore, the disadvantage of known media materials such as mica, aluminium oxide, bornitrite or beryllium oxide is that they are partly highly toxic (beryllium oxide, for example) and display a high specific thermal conductive resistance. This relates in particular to ceramic and polymeric foil systems. Viscous systems such as pastes, fats, paints, glues etc. can for the most part not be applied during the production process of the electronic components to be cooled and are therefore difficult to handle.

[0031] The use of adamantine carbon structures as electrical insulator of heat transfer media leads to considerably improved distinguishing and efficiency characteristics and guarantees optimum processing with the corresponding media support in adaptation to the respective components. At the same time the material according to the present invention represents an optimum electrical insulator and results in improvement of a series of other performance characteristics of the overall system, such as hardness, dielectric constant, breakdown load, specific

density, tensile strength, breaking strength, and cut-through resistance, expansion, loss factor etc.

[0032] The material according to the present invention can be doped for specific applications and lends itself to large-scale and cost-effective production. In addition, the technical reliability and service quality as well as the efficiency of running electronic components can be substantially increased by the elimination of optimised power loss without constructive changes being made to assemblies and structural components.

[0033] To obtain the distinguishing and efficiency characteristics adamantane carbon structures in solid highly dispersed form or as a suspension, dispersion or emulsion are added to corresponding support systems. According to the present invention the carbon structures thus added exhibit highly structured surfaces and forms which in particular enable a focused variation of the required cross-linking, suspension and dispersion properties, to guarantee optimum system characteristics.

[0034] Various examples of application of the method according to the present invention and for use of the adamantane carbon structures according to the present invention are illustrated hereinbelow and described in greater detail.

[0035] For dynamic chemical production of adamantane carbon structures an energy carrier such as 2, 4, 6 tri-nitro-toluol/cyclotrimethylene trinitramine (50/50)

of mass 0.5 kg with cylindrical form is introduced to the centre of a closed high-pressure reactor system (autoclave) with geometric dimensions in a ratio of L/H mod 8.6 to 9.2 (L = length, H = height) and R/H mod 4 to 6 (R = inner radius) in a volume of 3.0 m³. The chemical conversion of the energy carrier has to take place under light vacuum conditions (ca. 2 mm Hg), and is generated by a corresponding vacuum pump. The following processing parameters are to be complied with:

pressure plateau in chemical peak: P = 20 to 30 Gpa

temperature plateau in chemical peak: T symbol 179 \f "Symbol" \s 12 □
4.000 K

length of P/T plateau: T symbol 163 \f "Symbol" \s 12 □ 3 x 10⁻⁶ s

conversion rate: D symbol 179 \f "Symbol" \s 12 □ 8.32 x 10³ m/s.

[0036] The chemical reaction is initiated by means of an electrically produced thermal and dynamic impulse (electrodetonator).

Example 1

[0037] In compliance with the abovementioned technological guidelines an atomic hydrogen-supported low-temperature plasma is produced at 4000 K:

particle numbers in cm^3 : ^1H symbol 179 \f "Symbol" \s 12 $\square 9.4 \times 10^{17}$; ^1O symbol 179 \f "Symbol" \s 12 $\square 4.7 \times 10^{17}$

density [g/cm^3]: 2.3×10^{-5}

enthalpy [J/kg]: 3.4×10^4

specific heat [J/gK]: 32.5.

[0038] For the supporting chemical forming of cubic carbon structures a water vapour plasma is produced by a water receiver surrounding the energy support system under the effect of the abovementioned processing parameters (pressure, temperature, time). The thickness of the water receiver corresponds to three times the diameter of the cylindrical body of the energy support used.

[0039] The adamantine carbon structures formed as a result of this process are distinguished by the following parameters and characteristics:

process output (process yield) of adamantine carbon structures according to the present invention 10.43 % (relative to the used mass of the energy medium)

purity of the cubic crystal lattice phase: $2\theta = 100\%$, no proof of roentgen-amorphous phase portions (cf. Figure 5)

specific surface: $267.85 \text{ m}^2/\text{g}$

Zeta potential: $+10 \text{ mV}$

surface polarity: hydrophobic

other parameters corresponding to Figures 1 and 2.

Example 2

[0040] In compliance with the general technological guidelines as well as conditions for producing an atomic hydrogen-supported low-temperature water vapour plasma according to Example 1, before the chemical conversion reactions are initiated gaseous/liquid media mixtures such as for example ethylene/butane/propane in a volume ratio of 20/40/40 with a pressure level of the order of 0.3 Mpa to 0.8 Mpa are to be placed or blown into the high-pressure reactor system.

[0041] The distinguishing and efficiency characteristics of the produced adamantane carbon structures are changed as follows:

process output of carbon structures according to the present invention:

12.42 %

purity of the cubic crystal lattice phase: 100 %

specific surface: $298.85 \text{ m}^2/\text{g}$

Zeta potential: +5.6 mV

surface polarity: slightly hydrophobic

Example 3

[0042] In compliance with the abovedescribed conditions for producing an atomic hydrogen-supported low-temperature water vapour plasma according to this example, before the chemical conversion reactions are initiated liquid organic carbon donor systems of the aliphatic series, such as alkane or monovalent or polyvalent alcohols (ethylene glycol, for example) are introduced in the form of a sheathing between cylindrical bodies of the energy medium and water receiver to the system.

[0043] The mass (M_{KS}) of the carbon donor system is determined as follows:

$$M_{KS} = A \times M / 100 - A, [g]$$

[0044] A = % portion of the carbon donor system in the overall reaction system and M = energy medium mass + carbon donor mass, where A (%) must be of the order of 13.8 to 15.1.

[0045] This procedural step delivers adamantine carbon structures according to the present invention with mainly the following properties:

process output: 31.6 %

purity of cubic crystal lattice phase: 100 %

specific surface: 325.65 m²/g

Zeta potential: - 4.5 mV

surface polarity: slightly hydrophilic.

Example 4

[0046] In this embodiment all parameters of Example 3 are adhered to and unsaturated alcohols (such as for example propargyl alcohol or allyl alcohol) or aromatic compounds (such as for example benzol, nitrobenzol or acetonitrile) are used as carbon donor systems. The value of A (%) to be used here must be of the order of between 12.8 and 17.3.

[0047] The adamantine carbon structures to be produced thereby exhibit the following characteristics:

process output: 52.4 %

purity of cubic crystal lattice phase: 100 %

Zeta potential: + 10.0 mV

specific surface: 348.50 m²/g

surface polarity: strongly hydrophilic.

Example 5

[0048] In compliance with the generally technical-technological parameters an aqueous slurry of DLSC material (for example, produced using the technological steps of Examples 1 to 4) is set up in the high-pressure reactor unit in the form of a sheathing of the cylindrical energy carrier system and 5 to 8 % water (entionised). The mass of the slurry system must correspond at least to 1.35 times the mass of the energy carrier. The resulting adamantine carbon structures are presented in polycrystalline form (PK-DLSC) with typically spread X-ray pattern peak at (111), (220) and (311). The X-ray pattern is shown in Figure 6.

[0049] The crystallite sizes are of the order of 5 to 20 nm and are polycrystallised correspondingly by sp^3 hybridising. The available polycrystals with a 100 % purity of the cubic crystal lattice phase vary in grain size ranges of approximately 0.5 μm to 15 μm , whereby a median value of 3.43 μm and standard deviations of less than 2.41 μm can be achieved. Figure 7 shows the grain size distribution diagram. The process output of this technological method is ca. 50 %.

[0050] The following distinguishing and efficiency characteristics are achieved by the adamantine carbon structures of all Examples 1 to 5:

| | |
|--|---|
| Modification colour | light grey to black |
| Carbon phase portion, cubic | 98 - 100 % [(111)50/37 = 2 Θ] |
| Grain form - monocrystalline/cluster | ogival |
| Granulation range - monocrystalline/cluster | 5 nm - 10 μm 50 nm - 20 μm |
| Bulk density, average | 0.4 g/cm ³ |
| Density, pycrometric | 3.42 g/cm ³ |
| Surface, specific | 200-350 m ² /g |
| Magnetisation, specific | > 4 x 10 ⁻⁸ m ³ /kg |
| Zeta potential | - 15/+ 10 mV |
| Constituents, incombustible | < 2 % |
| Portions, volatile | < 3 % |
| | |
| Additives | |
| Hydrogen | < 0.9 % |
| Nitrogen | < 2.0 % |
| Oxygen | < 10.0 % |
| | |
| Thermal conductivity | 1.300-2.100 W/mK |
| Expansion coefficient, linear | 1.1-3.6 x 10 ⁻⁶ K |
| Oxidation temperature in air | > 400 °C |
| Graphitising temperature in vacuum | 1.100-1.250 °C |

[0051] Prototype 1

hydrophilised specific surface parameter

specific magnetising capacity: (- 0.4) Lambda x 10⁻⁸ m³/kg

specific electrical resistance: 1.6 x 10¹⁰ Ohm x m

free energy (water vapour absorption): - 973.2 mJ/g x Mol

[0052] Prototype 2

hydrophobised specific surface parameter

specific magnetising capacity: $(0.36) \text{ Lambda} \times 10^{-8} \text{ m}^3/\text{kg}$

specific electrical resistance: $6.6 \times 10^{10} \text{ Ohm} \times \text{m}$

free energy (water vapour absorption): $- 2.22 \text{ mJ/g} \times \text{Mol}$

[0053] The abovedescribed so-called DLSC materials in mono- and polycrystalline form represent a new generation of highly structured carbon systems whose general distinguishing and efficiency characteristics correspond to the cubic crystal lattice phase of the pure diamond and also exhibit a series of specific, chemical-physical properties which can be varied especially doped. From the morphological viewpoint the present material thus allows relevant technical interfaces to be realised for transfer to hyperstructured carbon systems, in particular to fullerene and hyper-fullerene in the form of onion-like carbons and nano-tubes.

[0054] With the addition of the abovementioned and illustrated properties the material can be used advantageously both in original form and also in conjunction with other materials (composites), as compared to known materials and conventional substances, particularly for the areas of application itemised hereinbelow:

abrasive and tribological systems for nano-high-finishing of high-quality
material surfaces

transfer and insulation systems

engineering composites

coating systems

selected biotechnical and genetechanical materials.

[0055] For surface treatment of hard and superhard materials the DLSC are to be placed in specially designed media. This is explained with reference to the following examples:

[0056] Example 1

Aqueous suspensions of sprays based on cationic short-chain suspension auxiliary products (dispersing and stabilising systems) with degrees of polyrisation of ca. 100 to 5000.

Suspension: DLSC – S/0,n.....,n

Contents:

- DLSC, monocrystalline, particle size classification 0,n.....,n .

: 0.1- 25.0 %

- Polydimethyl diallyl ammonium chloride

Sodium polyacrylate

Chitosan : 1.0 - 8.0 %

• Distilled water : % - residual portion

• pH value : 4 -12

Suspension: DLSC - S I / 0,n.....n

Contents:

• DLSC, polycrystalline, particle size classification 0,n.....n

: 0.1 -10.0 %

• Polydimethyl diallyl ammonium chloride : 2.0 4.0 %

• Distilled water : % - residual portion

• pH value : 4-12

Spray: DLSC - Sp / 0,n.....n

Contents: - as per (1) -

Carriers / propellants: polyglycol, propane, butane

Spray: DLSC - Spl / 0,nn

Contents: - as per (2) -

Carriers / propellants: - as per (3) -

Slurry: DLSC – PRM/0,n.....n

Contents:

• Composite graphite carbon/DSC (70/30) : 0.1 - 20 %

- Polydimethyl diallyl ammonium chloride : 1.0 - 8.0 %
- Distilled water : % -residual portion
- pH value : 4-12

Slurry: DLSC - SL/ 0,n1n1 / 0,n2.....n2

Contents:

- DLSC, monocrystalline, grain classes 0,n1n1
: 0.1-15 %
- Industrial diamond, monocrystalline, grain classes 0,n2.....n2
: 0.05 – 2.0 %
- Distilled water : % -residual portion
- pH value : 4-12

[0057] Example 2

DLSC – Emulsion based on water-soluble organic compounds

Emulsion: DLSC – E / 0,n.....n

Contents:

- DLSC, monocrystalline, grain classes 0,n.....n : 1.0 – 8.0 %
- Silicon oil AK 230 : 6.0 %
- Hoechst wax S : 5.0 %
- Olein : 0,7 %

- White spirit : 20.0 %
- Diethyl amino ethanol• : 1,3 %
- Ethylene glycol : 5.0 %
- Distilled water : % - residual portion

Emulsion: DLSC – EI / 0,n.....n

Contents:

- DLSC, polycrystalline, grain classes 0,n.....n
: 0,5 – 6.0 %

as per (1)

[0058] Example 3

DLSC - Pastes /waxes based on water-soluble organic compounds

Paste: DLSC - P / 0,nn

Contents:

- DLSC, monocrystalline, grain classes 0,n...,n: 4.0 –12 %
- Hoechst wax KSL : 5.0 %
- Stearic acid, technical : 4.0 %
- Petroleum : 10.0 %
- Citric acid : 5.0 %
- Potassium hydroxide (86 %) : 6.0 %

- Distilled water : % - residual portion

Paste: DLSC - P I /0,nn

Contents:

- DLSC, polycrystalline, grain classes 0,n.....n

: 1.0 – 10 %

- - as per (1)

[0059] Client uses for different areas of application relative to the best prior art are illustrated hereinbelow:

[0060] Thermal conducting systems such as foils, glues, grouts, pastes, solders, tapes

| System parameters | Added-Value-Factor [AVF] |
|---------------------------------------|--------------------------|
| Thermal conductivity | > 7 |
| Thermal resistance | > 5 |
| Dielectric constant [60 Hz] | > 2 |
| Dielectric constant [1 kHz] | > 3 |
| Temperature range (continuous duty) | 1.3 |
| Specific volume/throughput resistance | > 2.3 |
| Breakdown load | > 1.7 |
| Shore A hardness | > 1.3 |
| Specific density | < 1.8 |
| Tensile strength | > 2.6 |
| Breaking strength | 1.3 |
| Expansion | < 2 |
| Cut-through resistance | > 1.3 |
| Loss factor [60 Hz] | > 35 |
| Loss factor [1 kHz] | > 20 |

[0061] Substantial increase in chemical stability and strength as compared to gaseous and liquid aggressive media

[0062] Abrasive and tribological systems as suspensions, emulsions, waxes, sprays, desiccants ...

| System parameters | Added-Value-Factor [AVF] |
|---|--------------------------|
| Reduction of friction coefficient | > 1.9 |
| Obtaining coarseness qualities into the molecular range | < 5 - 10 nm |

[0063] Engineering ceramics, engineering polymers, tapes, coatings, nano-composites ...

| System parameters | Added-Value-Factor [AVF] |
|---|--------------------------|
| Abrasion resistance | > 1.8 |
| Friction coefficient | > 1.8 |
| Tensile stress at 300 % elongation | 1.9 |
| Expansion resistance | > 1.45 |
| Relative elongation | > 1.35 |
| Cracking resistance | > 2.4 |
| Increase of functional stability of elements, subassemblies and parts | 3 - 20 |

[0064] Surface coatings as powder, galvanic or polymer coatings

| System parameters | Added-Value-Factor [AVF] |
|------------------------|--------------------------|
| Microhardness | > 2 |
| Wear | > 1.8 |
| Friction against steel | > 12-30 |
| Stability | > 4-15 |
| Durability cycles | > 4-15 |

CLAIMS

1. A method for dynamic-chemical production of adamantine carbon structures,
 - wherein a hybrid carbon phase is placed into a closed container and
 - is brought to a chemical reaction with an energy support to form disperse condensed carbon as reaction product,characterised in that the reaction product is subjected to an atomic hydrogen-supported low-temperature plasma and a phase change from carbon combinations to very pure cubic lattice structures is made.
2. The method as claimed in Claim 1, characterised in that a carbon donor system is also introduced.
3. The method as claimed in Claim 2, characterised in that the carbon donor system has liquid or gaseous carbon compounds.
4. The method as claimed in Claim 2 or 3, characterised in that the carbon donor system has organic carbon compounds.
5. An adamantine carbon structure, characterised in that the purity of the cubic diamond phase is 99 % and preferably 100 %.

6. The adamantine carbon structure, characterised in that the crystallite sizes are of the order of between 5 nm and 50 nm.
7. The adamantine carbon structure, characterised in that the cluster orders of magnitude are between 50 nm and 20 μ m.
8. The adamantine carbon structure, characterised in that the particle diameters in dispersion are between 40 nm and 500 nm.
9. A use of adamantine carbon structures, in particular as claimed in any one of the foregoing claims for surface treatment of hard materials.
10. The use of adamantine carbon structures, in particular as claimed in any one of Claims 1 to 8, as electrical insulator.
11. The use of adamantine carbon structures, in particular as claimed in any one of Claims 1 to 8, as thermal transfer medium.
12. The use of adamantine carbon structures, in particular as claimed in any one of Claims 1 to 8, as addition to a suspension, dispersion, emulsion, a spray, a paste, a fat or a wax.

ABSTRACT

A hybrid carbon phase, which is brought to a chemical reaction with an energy carrier, is placed into a closed container for dynamic-chemical production of adamantane carbon structures to obtain disperse condensed carbon as reaction product. This reaction product is subjected to an atomic hydrogen-supported low-temperature plasma, bringing about a phase change from carbon to in very pure cubic lattice structures.

This produces adamantane carbon structures which have a purity of the cubic diamond phase of approximately 100 %. The crystallite sizes are of the order of between 5 nm and 50 nm and the cluster orders of magnitude are between 50 nm and 20 μm . The particle diameters in dispersion are between 40 nm and 500 nm.

The carbon structures according to the present invention are suited to surface treatment of hard materials, as electrical insulator or as thermal transfer medium.

For this purpose the adamantane carbon structures are added to a suspension, a dispersion, an emulsion, a spray, a paste, a fat, a wax or a paint system.

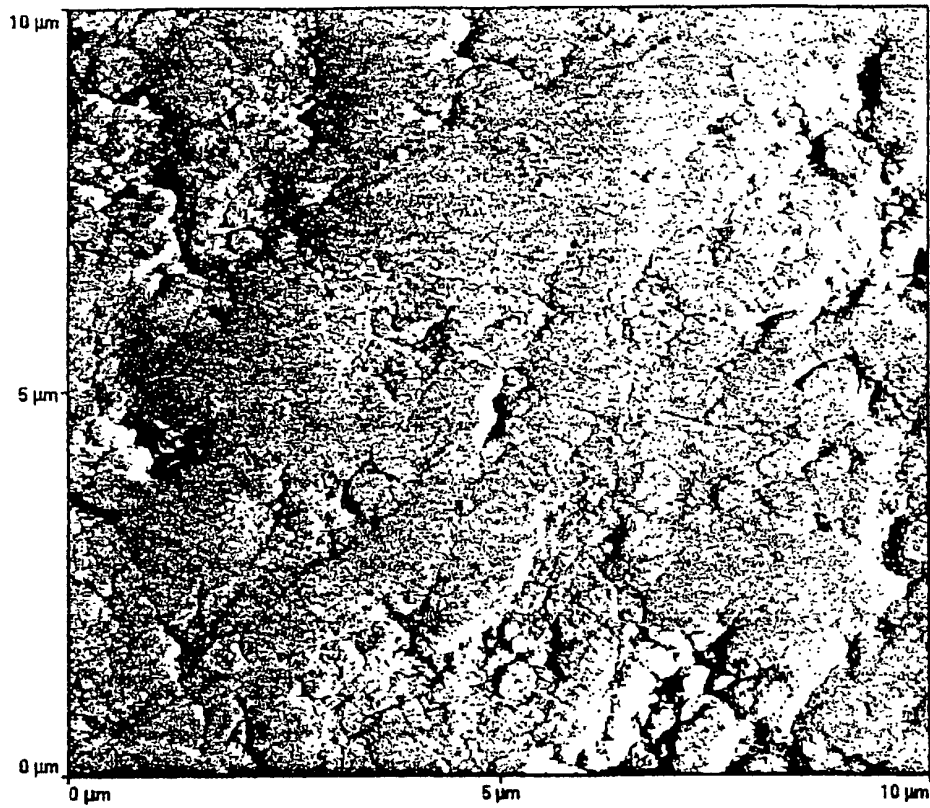


Fig. 1

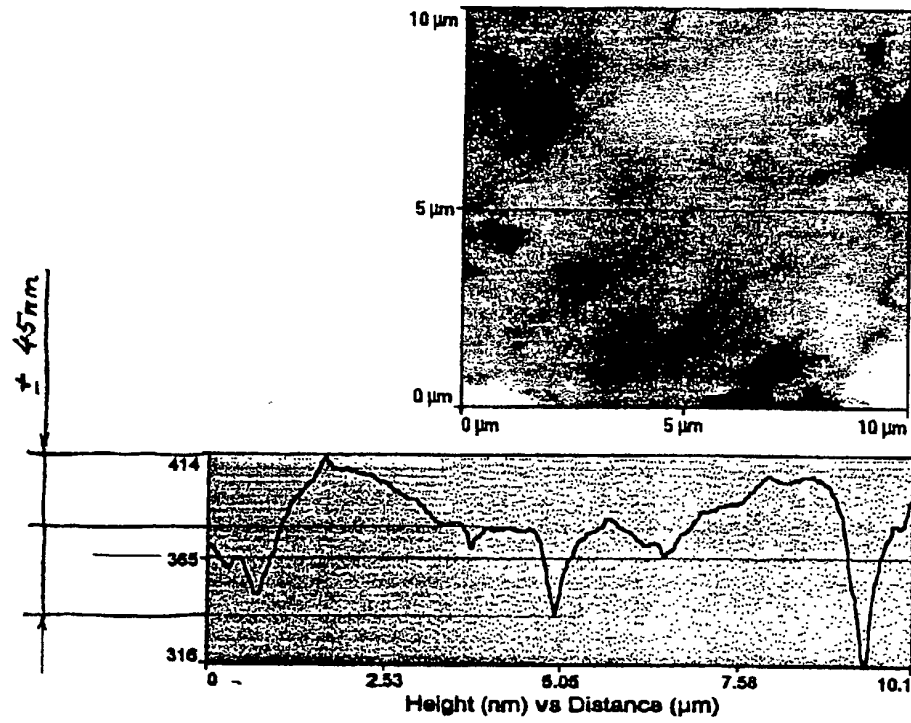


Fig. 2

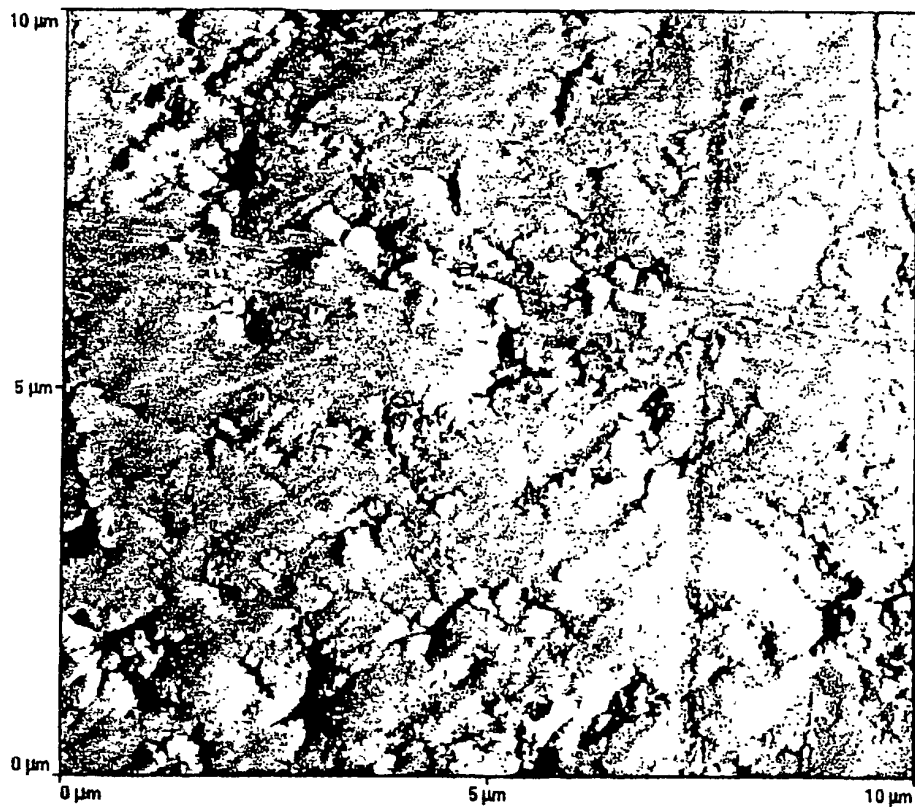


Fig. 3

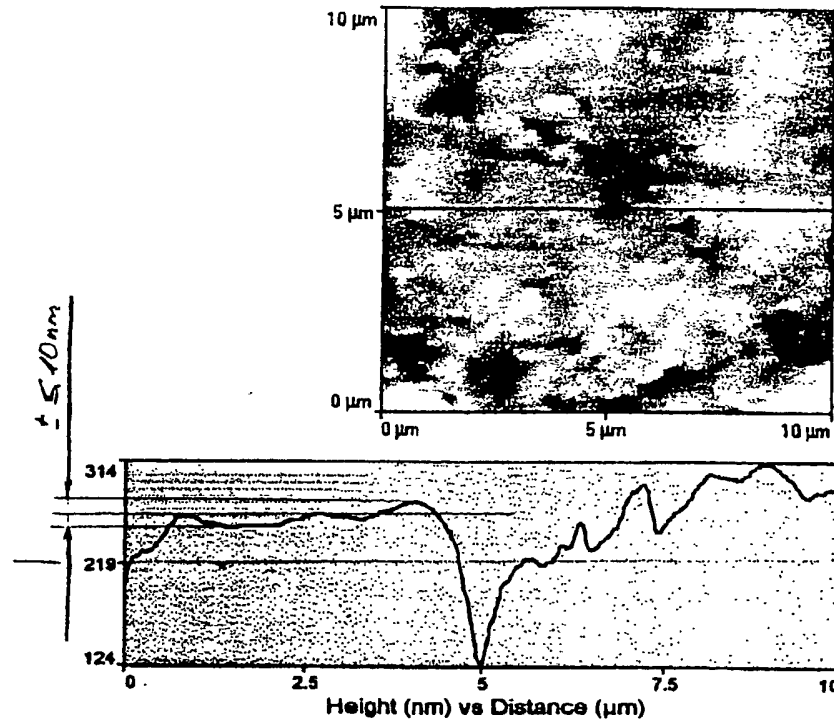


Fig. 4

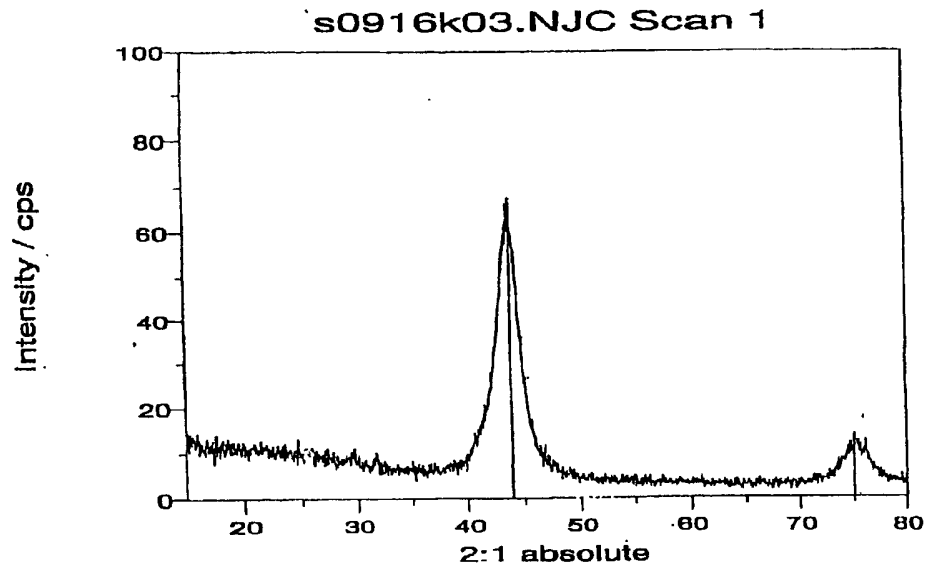


Fig. 5

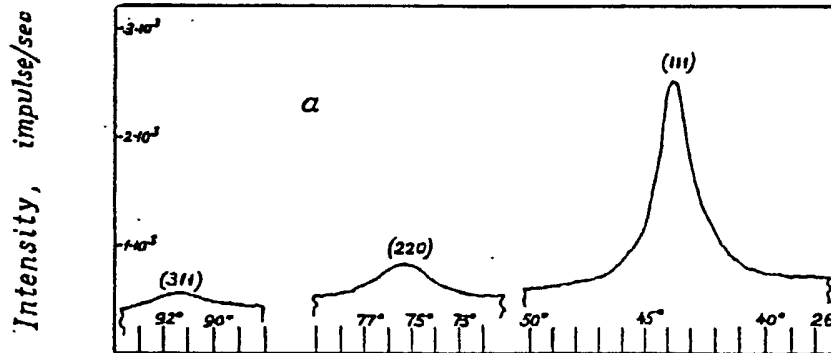


Fig. 6

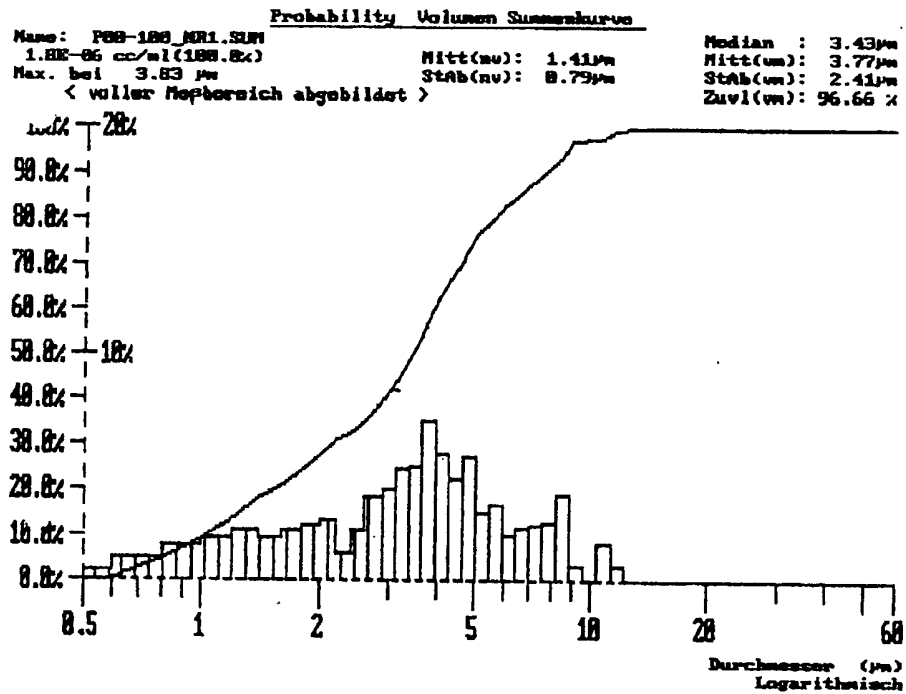


Fig. 7

Declaration and Power of Attorney for Patent Application

Erklärung für Patentanmeldungen mit Vollmacht

German Language Declaration

Als nachstehend benannter Erfinder erkläre ich hiermit an Eides Statt:

daß mein Wohnsitz, meine Postanschrift, und meine Staatsangehörigkeit den im Nachstehenden nach meinem Namen aufgeführten Angaben entsprechen,

daß ich, nach bestem Wissen, der ursprüngliche, erste und alleinige Erfinder (falls nachstehend nur ein Name angegeben ist) oder ein ursprünglicher, erster und Miterfinder (falls nachstehend mehrere Namen aufgeführt sind) des Gegenstandes bin, für den dieser Antrag gestellt wird und für den ein Patent beantragt wird für die Erfindung mit dem Titel:

VERFAHREN ZUR DYNAMISCH-CHEMISCHEN HERSTELLUNG VON DIAMANTARTIGEN KOHLENSTOFFSTRUKTUREN, DIAMANTARTIGE KOHLENSTOFFSTRUKTUREN UND VERWENDUNGEN VON DIAMANTARTIGEN KOHLENSTOFFSTRUKTUREN

deren Beschreibung
(zutreffendes ankreuzen)

- ☒ hier beigelegt ist.
☐ wurde angemeldet am 14. Juni 2000
 unter der U.S.-Anmeldungs Nr. oder unter der Internationalen Anmeldenummer im Rahmen des Vertrags über die Zusammenarbeit auf dem Gebiet des Patentwesens (PCT)
PCT/DE00/01885 und am _____
 abgeändert (falls zutreffend).

Ich bestätige hiermit, daß ich den Inhalt der obigen Patentanmeldung einschliesslich der Ansprüche durchgesehen und verstanden habe, die eventuell durch einen Zusatzantrag, wie oben erwähnt, abgeändert wurde.

Ich erkenne meine Pflicht zur Offenbarung irgendwelcher Informationen an, die für die Prüfung der vorliegenden Anmeldung in Einklang mit Titel 37, Code of Federal Regulations, §1.56 von Belang sind.

Ich beanspruche hiermit ausländische Prioritätsvorteile gemäss Titel 35, US-Code, §119(a)-(d), bzw. §365(b) aller unten angegebenen Auslandsanmeldungen für ein Patent oder Erfinderurkunden, oder §365(a) aller PCT internationalen Anmeldungen, welche wenigstens ein Land ausser den Vereinigten Staaten von Amerika benennen, und habe nachstehend durch ankreuzen sämtliche Auslandsanmeldungen für Patente oder Erfinderurkunden oder PCT internationale Anmeldungen angegeben, deren Anmeldetag dem der Anmeldung, für welche Priorität beansprucht wird, vorangeht.

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

VERFAHREN ZUR DYNAMISCH-CHEMISCHEN HERSTELLUNG VON DIAMANTARTIGEN KOHLENSTOFFSTRUKTUREN, DIAMANTARTIGE KOHLENSTOFFSTRUKTUREN UND VERWENDUNGEN VON DIAMANTARTIGEN KOHLENSTOFFSTRUKTUREN

the specification of which
(check one)

- ☒ is attached hereto
☐ was filed on June 14, 2000
 as United States Application Number or PCT International Application Number
PCT/DE00/01885, and was amended on _____
 (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Applications
(Frühere ausländische Anmeldungen)

Priority Claimed?
Priorität beansprucht?

| | | | | |
|---|---------------------------------------|---|--|--|
| <u>199 27 895.4</u> (Number) (Nummer) | <u>Germany</u> (Country) (Land) | <u>18/June/1999</u> (Day/Month/Year Filed) (Tag/Monat/Jahr eingereicht) | <input checked="" type="checkbox"/> [X] Yes Ja | <input type="checkbox"/> [] No Nein |
| <u>199 27 894.6</u> (Number) (Nummer) | <u>Germany</u> (Country) (Land) | <u>18/June/1999</u> (Day/Month/Year Filed) (Tag/Monat/Jahr eingereicht) | <input checked="" type="checkbox"/> [X] Yes Ja | <input type="checkbox"/> [] No Nein |
| <u>199 27 893.8</u> (Number) (Nummer) | <u>Germany</u> (Country) (Land) | <u>18/June/1999</u> (Day/Month/Year Filed) (Tag/Monat/Jahr eingereicht) | <input checked="" type="checkbox"/> [X] Yes Ja | <input type="checkbox"/> [] No Nein |
| <u>199 55 971.6</u> (Number) (Nummer) | <u>Germany</u> (Country) (Land) | <u>19/November/1999</u> (Day/Month/Year Filed) (Tag/Monat/Jahr eingereicht) | <input checked="" type="checkbox"/> [X] Yes Ja | <input type="checkbox"/> [] No Nein |

Ich beanspruche hiermit gemäss Titel 35, US-Code, §119(e), den Vorzug aller unten aufgeführten US-Hilfsanmeldungen

I hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) below

(Application No. / Anmeldenr.)

(Filing Date / Anmeldedatum)

(Application No. / Anmeldenr.)

(Filing Date / Anmeldedatum)

Ich beanspruche hiermit gemäss Titel 35, US-Code, §120, den Vorzug aller unten aufgeführten US-Patentanmeldungen bzw. §365(c) aller PCT internationalen Anmeldungen, welche die Vereinigten Staaten von Amerika benennen, und erkenne, insofern der Gegenstand eines jeden früheren Anspruchs dieser Patentanmeldung, bzw. PCT internationalen Anmeldung in einer gemäß dem ersten Absatz von Titel 35, US-Code §112 vorgeschriebenen Art und Weise offenbart wurde, meine Pflicht zur Offenbarung jeglicher Informationen an, die zur Prüfung der Patentfähigkeit in Einklang mit Titel 37, Code of Federal Regulations, §1.56 von Belang sind und im Zeitraum zwischen dem Anmeldedatum der früheren Patentanmeldung und dem nationalen oder im Rahmen des Vertrags über die Zusammenarbeit auf dem Gebiet des Patentwesens (PCT) gültigen internationalen Anmeldedatum bekannt geworden sind.

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s), or §365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

(Appl. No.)
(Anmeldenr.)

(Filing Date)
(Anmeldedatum)

(Status)
(patentiert, anhängig
aufgegeben)

(Status)
(patented, pending
abandoned)

(Appl. No.)
(Anmeldenr.)

(Filing Date)
(Anmeldedatum)

(Status)
(patentiert, anhängig
aufgegeben)

(Status)
(patented, pending
abandoned)

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

HENRY M. FEIEREISEN
Reg. No. 31,084

URSULA B. DAY
Reg. No. 47,296

Telefongespräche bitte richten an:
(Name und Telefonnummer)

Direct telephone calls to:
(Name and telephone number)

HENRY M. FEIEREISEN
(212) 244-5500

Postanschrift:

Send Correspondence to:



1-00
Voller Name des einzigen oder
ursprünglichen Erfinders

Full name of first inventor

CHRISTA SCHÖNEFELD

Unterschrift des Erfinders

Datum

Inventor's Signature

Date

+

23.01.2002

Wohnsitz

Geilenkirchen / Germany

DEX

Residence

Staatsangehörigkeit

Germany

Citizenship

Postanschrift

Heinrich-Zille-Weg 2
52511 Geilenkirchen
Germany

Post Office Address

Voller Name des zweiten Erfinders

Full name of second inventor

TEJAS SCHÖNEFELD

2-00
+

Datum

Inventor's Signature

Date

23.01.2002

Wohnsitz

Wassenberg / Germany

DEX

Residence

Staatsangehörigkeit

Germany

Citizenship

Postanschrift

Ring Strasse 22
41849 Wassenberg
Germany

Post Office Address